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The Colorimetric Determination of "Porphyroxine-Meconidine".

Communication on Experiments to Find a Relation Between "Red Lovibond Units" and Absorption Density Value of the Red "Porphyr" Solution

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In continuation of the international programme of scientific research undertaken pursuant to resolutions 159 II C (VII) and 246 F (IX) adopted by the Economic and Social Council on 3 August 1948 and 6 July 1949 respectively, the Secretary-General is presenting the following paper contributed by collaborating scientists of Austria. The paper establishes a correlation between photometer readings and the red Lovibond units used by the Secretariat, so that those who do not have the Lovibond may be able to interpret colorimetric readings for "porphyroxine-meconidine" in determining the origin of opium.

The Colorimetric Determination of "Porphyoxine-Meconidine"

Communication on Experiments to Find a Relation Between "Red Lovibond Units" and Absorption Intensity Values of the Red "Porphyr" Solution

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1. Purpose of the paper: the documents E/CN.7/195, ST/SOA/SER.K/4 and all others issued by the United Nations about "porphyroxine-meconidine" give the figures for intensity of the red colour in "red Lovibond units". As most interested laboratories in Europe do not possess a Lovibond Tintometer it will not be possible for them to make direct use of the research work on "porphyroxine-meconidine" done by the United Nations Secretariat. Moreover, we see no advantage in measuring with undefined, conventional units, where the reproducible extinction values for a certain wavelength could be used. For these reasons we have tried to work out a key for converting the "red Lovibond units" into extinction values and vice-versa.

2. Our determinations were carried out according to E/CN.7/195 on 27 of the samples listed in ST/SOA/SER.K/4. The absorption intensity of the red colour was measured with the Pulfrich Stufenphotometer of Zeiss using the green spectral filter S 53 (maximum of permeability 528 mμ, effective centre 530 mμ), and some comparative readings were made with a Beckman Photometer. The filter was chosen after the absorption-curve had been determined by means of the Beckman Spectrophotometer, and a suitable maximum had been found at the wavelength of 520 mμ. To find a relation not only in regard to values obtained by instruments with the above mentioned filter, but also to values obtained by photoelectric instruments with strict monochromatic light, we determined the absorption intensities of ten samples with the Beckman Spectrophotometer at the maximum of 520 mμ. The results of the Beckman readings proved to be higher, and the reason is clear from the absorption curve. It shows a rather steep decline after the 520 mμ maximum, thus explaining the lower values at the effective centre of 530 mμ of the S 53 filter.

3. As no calibration-curve is available to give any idea of the absolute quantity of "porphyroxine-meconidine", we think it the more important to make possible a comparison of the colour intensities.

/4. Suitable

4. Suitable concentrations of the red solution for photometer readings were obtained when the volume was made up to 100 ml. In practice, the colour was developed, brought to 10 ml with 5 volume-per cent acetic acid, and 1 ml of this solution was again diluted to 10 ml. This dilution (1 per cent w/v referred to opium) proved to be suitable for measurement in the 1 cm cell of the Beckman and the Zeiss instruments. In the case of samples with extremely high values the 1/2 cm cell of the Zeiss Stufenphotometer was used. Only extremely low values require the first dilution (10 per cent w/v referred to opium) for the determination, and in our experiments this was necessary in one case only (opium Korea No. 1, B, Lov. value 2.5). For this reason all absorption intensities are given in our Table in terms of the coloured solution of 100 ml from 1 g opium and in the 1 cm cell; in the following referred to as $E^{1}_{1\ cm}$ per cent w/v.

5. The fact that in nearly all cases the 1 g: 100 ml dilution could be used and that for the colorimetric determination 10 ml of the red solution is quite sufficient, gave us the idea of developing a semi-micro method with 0.15 g opium, on which we are now working.

6. It seemed very important to us to take a sample of average composition for the determination. This can hardly be done, if the fresh opium is used. Most of the samples are inhomogeneous, covered with varying envelopes, and the moisture-content in the outer parts of a loaf is lower than inside. The opium samples of the United Nations were therefore prepared as follows: the envelope (poppy leaves, paper, Rumex fruits, etc.) was removed, the lump reduced to small pieces and dried in a vacuum-exsiccator for five days. The loss of weight ranged from 2.11 to 5.88 per cent, being an average of 3.99 per cent. After this the opium could easily be powdered and was stored in glasses sealed with paraffin. For the determination a quantity of this dried and powdered opium was used which was equal to 1.5000 g fresh opium.

7. In the attached table we present the results of our studies. The first column gives the absorption intensities obtained with the Zeiss Stufenphotometer under the conditions described above, the samples being listed in the order of increasing extinction-values. The correlation between the $E^{1}_{1\ cm}$ per cent w/v values obtained by us (column 1) and the Lovibond units (column 3) considered for all samples, gave an average of 0.0251 $E^{1}_{1\ cm}$ per cent w/v per 1 red Lovibond unit.

/One sample

One sample ("Yugoslavia No. 2") was not considered for this average, as no loaf was stated on the label. Ten out of the 26 samples showed a deviation of more than ± 10 per cent from this average value. These are marked with an asterisk in column 4. To be sure that these large deviations were not due to errors in the determination, a second determination of these ten samples was carried out and it showed no higher deviation than ± 1.5 per cent from the middle value. After that it was clear that the differences in the results were caused by the nature of the opium (other loaves, varying moisture, different samples, etc.). For this reason, the mentioned ten samples were left out of consideration in setting up the final correlation between the Lovibond units and the absorption intensities. The final average result from the remaining 16 samples was:

$E^{1}_{1\ cm}$ per cent w/v per 1 red Lovibond unit = 0.0253

Pulfrich Stufenphotometer Zeiss, filter S 53.

To find the correlation to Beckman Photometer values, ten samples of an average of 0.0253 $E^{1}_{1\ cm}$ per cent w/v (Zeiss Photometer) per Lovibond unit were measured with the Beckman Photometer. The values at the maximum of 520 mμ were found to be 13.13 per cent higher than the Stufenphotometer values on the average. We can therefore establish the correlation:

$E^{1}_{1\ cm}$ per cent w/v per 1 red Lovibond Unit = 0.0286

Beckman Spectrophotometer, model DU, 520 mμ.

9. Conclusion: in the hope of establishing a uniformly accurate correlation between Lovibond units and absorption intensities we were disappointed, as our United Nations samples showed deviations from the values given in ST/SOA/SER.K/4 up to ± 28 per cent. But as we left the samples with very high deviations out of consideration and took the average of the remaining samples, the figures given above can well be taken, in general, for the correlation between red Lovibond units and absorption intensities. We hope that our results will be a help to institutions which want to carry out examinations concerning the origin of opium and do not possess a Lovibond Tintometer. To establish an accurate "calibration" of the different colorimetric equipments it would be best to distribute a well mixed, dried, and powered opium with a well-defined Lovibond value as a standard to all interested laboratories.

/TABLE

TABLE

Opium	$E^{1}_{1\,cm}$ per cent Stufenphotometer, filter S 53	2nd determination	Lovibond Units	$E^{1}_{1\,cm}$ per cent per 1 red Lovibond Unit	
Korea No. 1	0.067		2.5	0.0268	
Turkey No. 4, Çal (B)	0.219		8.0	0.0273	
" No. 6, Nallihan (B)	0.236		10.0	0.0236	
" No. 7, Merzifon (B)	0.255	0.257	9.0	0.0283	*)
Greece (d)	0.258	0.264	12.8	0.0202	*)
Korea No. 2	0.288		11.6	0.0248	
Turkey No. 3, Çorum (B)	0.313	0.308	10.0	0.0313	*)
Indochina, Laokay (k)	0.312	0.321	13.8	0.0226	*)
Yugoslavia No. 1	0.333		12.4	0.0268	
China (B)	0.336		13.5	0.0249	
Turkey No. 8, Burdur (B)	0.370	0.373	18.0	0.0205	*)
" No. 15, Exportation	0.376	0.380	11.6	0.0324	*)
" No. 13, Isparta	0.387		16.0	0.0242	
" No. 11, Akhsehir (B)	0.438	0.439	22.4	0.0196	*)
" No. 2, Afyon	0.452		18.0	0.0250	
Indochina Xieng Khouang (B)	0.458		19.2	0.0239	
Turkey No. 5, Usak (B)	0.460		18.4	0.0250	
Indochina Luang Prabang (B)	0.474		20.0	0.0237	
Turkey No. 1, Aydin (C)	0.484	0.495	17.2	0.0281	*)
Yugoslavia No. 2 (C)†	0.534		21.8†	0.0245†	
Turkey No. 14, Malatya (C)	0.586		23.2	0.0253	
" No. 9, Kula (B)	0.602		23.9	0.0252	
India No. 3, Excise	0.749	0.755	27.0	0.0277	*)
Turkey No. 10, Sandikli (B)	0.852		32.6	0.0261	
India No. 2, Malwa	0.894		35.8	0.0250	
" No. 1, Banares	0.920		34.6	0.0266	
Turkey No. 12, Zile (C)	1.168	1.176	64.0	0.0182	*)

Figures of this table obtained by Pulfrich Photometer Zeiss, filter S 53.
*) marks the samples with a deviation of more than ± 10 per cent from the average value.

† As mentioned in the text, this sample was not used in computing the equivalent, because the loaf sent was not marked on the label. The loaf designation (c) and the Lovibond value have been supplied by the Secretariat from their records.